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Pulsed Photothermal Radiometry for Remote Sensing of Spectroscopic and Physical Properties of Thin Film

by

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San Jose, California

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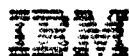
Research Report

PULSED PHOTOTHERMAL RADIOMETRY FOR REMOTE SENSING OF SPECTROSCOPIC AND PHYSICAL PROPERTIES OF THIN FILM

A. C. Tam

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ABSTRACT: Photothermal Radiometry (PTR) is a sensitive technique for noncontact spectroscopy and inspection; a modulated beam of photons (or other particles) is used to produce temperature transients, and the corresponding transients in the infrared thermal radiation emitted from the sample are analyzed. This paper describes the recent experimental and theoretical developments of pulsed PTR (PPTR) with back-scattering detection. PPTI is useful for many types of noncontact measurements, including absolute absorption spectroscopy, excitation spectroscopy, and the monitoring of thickness, layered structure, thermal diffusivity, thermal contacts, and associated properties. Furthermore, these measurements can be made on "difficult" samples like opaque materials, powders, aerosols, gels, skins *in-vivo*, and so on.

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The photo-thermal (PT) effect is the conversion of electro-magnetic energy, in part or in full, into heat energy in a material. PT effect can be detected directly, e.g., by "calorimetry",[1-4] or indirectly, e.g., by the photo-acoustic effect,[5-7] monitoring refractive index variations causing defocusing[8,9] or deflection[10-14] of probe beams, surface distortions,[15,16] spectroscopy[17] and radiometry. This paper is concerned with the last technique, i.e., photothermal radiometry (PTR). PTR relies on the detection of variations in the infrared (IR) thermal radiation emitted from a sample that is excited by electromagnetic radiation (typically from a laser or from an arc lamp) of varying intensity or wavelength. The advantages of PTR compared to other PT monitoring techniques are the following: (1) It is totally noncontact, so that measurements can be made on "difficult" circumstances, like vacuum, high pressure, high temperature, or other hostile environments. (2) With the use of IR detectors of fast rise time, thermal or mechanical properties of thin film samples can be measured in much shorter times (or even in "real time") compared to conventional methods. (3) Absolute values for absorption coefficients, thermal diffusivity, or thickness of samples can be obtained in a noncontact manner. (4) PTR performed in the back-scattered mode with pulsed laser for excitation appears to be useful for single-ended remote-sensing of samples that may be ~km away, i.e., back-scattering PTR may provide a new PT LIDAR (LIght Detection And Ranging).

There are four variations of PTR techniques that have been reported in the literature as indicated in Table I. These variations can be classified according to the excitation mode

TABLE I

Examples of Various Modes of Photothermal Radiometry

Source	Detection	
	Transmission	Back-scattering
Continuous modulated	Cowan[19] (1961)	Henuier and Hardy[26] (1961)
	Busse[20] (1980)	Nordal and Kanstad[27-29] (1979,80,81)
	Busse and Eyerer[21] (1983)	Luukkala[32] (1980)
	Busse and Renk[22] (1983)	Kanstad et al.[30,31] (1982,83)
		Vanzetti and Traub[33,34] (1983)
Pulsed	Parker et al.[24] (1961)	Tam and Sullivan[35] (1983)
	Deem and Wood[13] (1962)	Leung and Tam[36,37] (1984)
	Taylor[25] (1972)	Cielo[38] (1984)
		Imhof et al.[39] (1984)

(continuously modulated or pulsed) and to the detection mode (transmission or back-scattered). The excitation beam (which can be photon, electron, microwave, etc.) is usually either continuous modulated with about 50% duty cycle, or pulsed modulated with very low duty cycle, but high peak power. The detection can in principle be in any direction but is simpler for observation backwards from the excitation spot (called back-scattering PTR here), or "end-on" through the sample thickness with respect to the excitation spot (called transmission PTR here). Most workers have used either the back-scattering geometry or the transmission geometry, although Luukkala et al.[18] have demonstrated PTR with the observation spot being laterally displaced from the excitation spot. Only back-scattering PTR (not transmission PTR) can be used for thick or bulky materials, for samples with inaccessible back surface, or for single-ended remote sensing.

This paper is mainly concerned with the new technique of pulsed PTR (PPTR) in back-scattering, as indicated in Fig. 1. Various pulsed excitation sources can be used, for example a short-duration (full width at half maximum=8 nsec) N_2 laser beam with less than 1 mJ energy at 337 nm. The back-scattered PPTR measurement is shown for beam path (a) in Fig. 1; some of our work is also performed with transmission PPTR as indicated in beam path (b). The thermal IR radiation from the sample is refocused onto a HgCdTe detector with a rise time of 0.5 μsec, and the PPTR signal in back-scattering ($S_B(t)$) or in transmission ($S_T(t)$) is accumulated on a transient recorder (Tektronix 7854 scope with 7D20 plug-in). Maximum
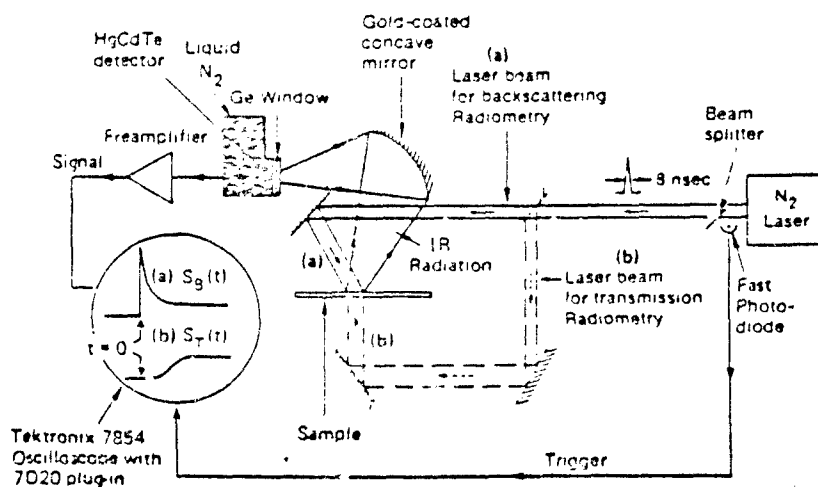

Fig. 1. Experimental setup for both single-ended back-scattering (laser beam represented by solid lines) and double-ended transmission (laser beam represented by dash lines) pulsed photothermal radiometry measurements.

sample heating by the laser beam of a spot size being several mm is generally limited to ~10°C to avoid any sample damage or any nonlinear radiometric effects (*i.e.*, the radiometric signal magnitude is proportional to the surface temperature rise ΔT only for small ΔT).

We[35] have applied this PPTR in back-scattering for novel absolute absorption spectroscopy of opaque solids and liquids. This is possible because the magnitude of the absorption coefficient α of the excitation beam is related to the "steepness" of the initial temperature profile produced in the sample. Large α corresponds to a steeper temperature profile near the sample surface, causing faster cooling of the surface and hence faster decay of the back-scattered PPTR signal. By analyzing the shape of the PPTR signal from bulk samples, Tam and Sullivan[35] showed that α can be obtained under some conditions if the thermal diffusivity D is known.

Another very important application[35] is the remote sensing of the layered structure of a sample by PPTR. To show this, we coat a black rubber substrate with a 45 μm thick polyester film that is transparent in the wavelength of the excitation beam. For this layered structure, the pulsed PTR exhibits two peaks: a prompt peak that decays with the same rate as the bare substrate but with decreased magnitude, and a delayed peak occurring at a time t_d after the firing of the laser. We found that t_d is related to the thickness ℓ and the thermal diffusivity D_ℓ of the coating by $t_d = \ell^2/4D_\ell$, indicating that the prompt peak is due to IR thermal radiation from the irradiated substrate and attenuated by the coating, and the delayed peak is due to IR thermal radiation from the top coating surface due to heat diffusion.

We have also[35] demonstrated that PPTR is useful for sensing the powder aggregation which affects the "effective" thermal conductivity. The pulsed laser is used to irradiate black carbon-loaded epoxy powders of different degree of compactness. We find that the PPTR signal for the loose powder stops decaying after some time indicating that inter-particle heat transport is slow, but the PPTR signal for a compact powder exhibits a continuous decay as in a neat solid.

We have investigated[36,37] the PPTR signal profile. For a sample of thickness L, thermal diffusivity D, absorption coefficient α at the excitation wavelength and α' at the detection wavelength, the PPTR signal shape excited by a pulsed laser of duration τ_0 is dependent on different parameters at different delay time periods (see Fig. 2). For simplicity in the present discussion, we assume that τ_0 is short, and the IR detector rise-time is fast. The theoretical PPTR signals $S_B(t)$ and $S_T(t)$ are given by

$$
\begin{bmatrix} S_B(t) \\ S_T(t) \end{bmatrix} = \frac{AK}{L} \left\{ (1-e^{-\alpha L})(1-e^{-\alpha' L}) \right.
$$

$$
\left. \pm 2 \begin{bmatrix} 1 \\ e^{-\alpha' L} \end{bmatrix} \sum_{n=1}^{\infty} \left(\frac{1-(-1)^n e^{-\alpha L}}{1+\frac{n^2\pi^2}{\alpha^2 L^2}} \right) \left(\frac{1-(-1)^n e^{\mp \alpha' L}}{1+\frac{n^2\pi^2}{\alpha'^2 L^2}} \right) e^{-n^2 t/\tau_L} \right\} \tag{1}
$$

Excitation

(a) t = 0

(b) $t \ll \tau_L$
$(\tau_L = L^2/\pi^2 D)$

(c) $t > \tau_L$
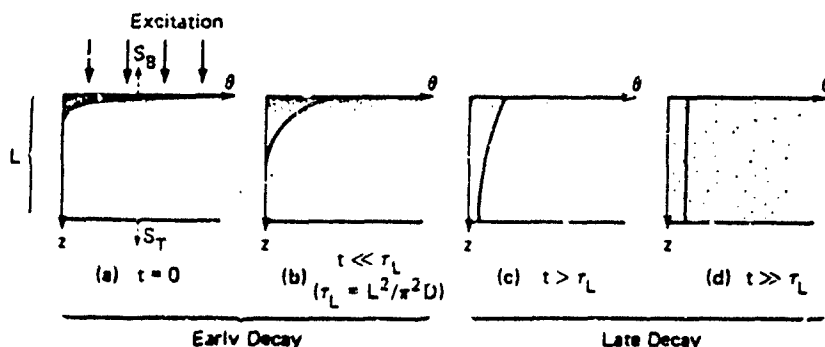
(d) $t \gg \tau_L$

Early Decay

Late Decay

Fig. 2. Thermal diffusion in an opaque sample of thickness L after a short pulsed heating at a surface. The local temperature increase above ambient (θ) is plotted at several times (t), and indicated by the density of the black dots. Early decay is spectroscopically related and late decay is dimension related.

where the equation for the upper/lower qualities in the two square brackets correspond to the upper/lower ± signs. Here, A is a constant depending on the flash pulse energy and the thermal properties, and K is a constant depending on the emissivity and the ambient temperature.

Some results based on Eq. (1) for $S_B(t)$ are shown in Figs. 3 and 4. Figure 3 shows the theoretical shapes of $S_B(t)$ for two samples of identical material (stainless steel) but different thickness: L=0.005cm and L=∞. We see that the two signals totally overlap at early times; however, after a "thickness diffusion time" τ_L=63.3 μsec for the L=0.005cm sample, the two signals start to deviate: the thin-film signal stops decaying while the "thick plate" signal continues to decay. These effects have indeed been verified experimentally[36,37] Figure 4 shows how the theoretical shapes of $S_B(t)$ depend on the absorption coefficients α and α', with fixed thickness and thermal diffusivity; at early times (i.e., $t<<\tau_L$), the stronger absorption coefficients cause a steeper decay of the signal. However, at late times ($t>>\tau_L$) the decay curves for different values of α and α' are overlapping, showing that the late decay rate is only dependent on L and D, in agreement with the analysis of Parker et al.[24] To sum up, the back-scattered PFTR signal $S_B(t)$ is sensitively dependent on α, α' and D for $t<<\tau_L$, and sensitively depend on L and D for $t>>\tau_L$. Theoretical fitting[37] of the observed $S_B(t)$ for a sample can provide values of α, α', D, and L. Such measurements have the distinct advantages of being single-ended, remote-sensing, and nondestructive. In Eq. (1), we have neglected the effects due to the finite excitation pulse width and the finite IR detector rise time. Such effects can be taken into account.[37]

Equation (1) approaches simple limits[37] when L is large. In this case, $S_T(t) \to 0$, and

$$S_B(t) \underset{L \to \infty}{\to} \frac{AK\alpha\alpha'}{\alpha'^2 - \alpha^2} \left\{ \alpha' e^{t/4\tau_\alpha}(1 - \mathrm{erf}\sqrt{t/4\tau_\alpha}) - \alpha\, e^{t/4\tau_{\alpha'}}(1 - \mathrm{erf}\sqrt{t/4\tau_{\alpha'}}) \right\} \qquad (2)$$

where $\tau_\alpha = (4\alpha^2 D)^{-1}$, $\tau_{\alpha'} = (4\alpha'^2 D)^{-1}$ and erf is the error function.[40] Equation (2) is symmetrical with respect to the absorption coefficients α and α'. It further simplifies if one of the absorption coefficient is much larger than another, e.g., $\alpha'>>\alpha$. In this case:

$$S_B(t) \underset{\substack{L \to \infty \\ \alpha'>>\alpha}}{\to} AK\alpha e^{t/4\tau_\alpha}(1 - \mathrm{erf}\sqrt{t/4\tau_\alpha}) , \qquad (3)$$
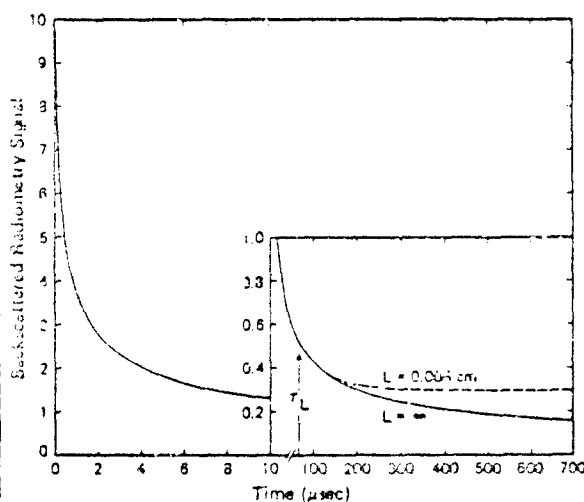

Fig. 3. Theoretical profiles of the single ended back-scattered flash radiometry signal showing the effect of sample thickness L. Dash line is for L=0.005 cm and solid line is for L=∞. In both curves, α, α' and D are taken as 2×10⁴ cm⁻¹, 1×10⁴ cm⁻¹ and 0.04 cm²/s, respectively, which are typical values for stainless steel. Note that the scale changes for the late decay in the inset.
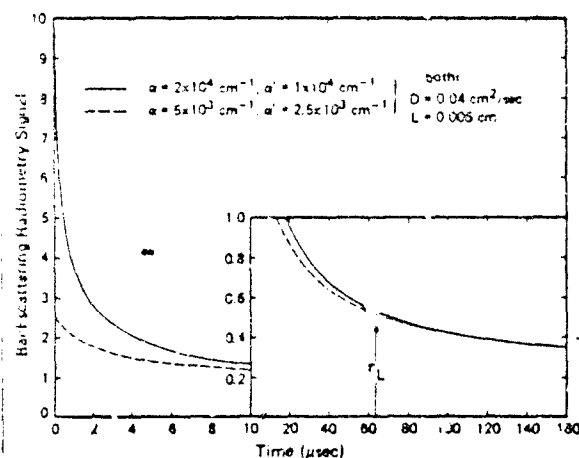
Fig. 4. Theoretical profiles of the back-scattered radiometry signal showing the effect of absorption coefficients α and α'. Dash lines is for α=5×10³ cm⁻¹ and α'=2.5×10³ cm⁻¹; solid line is for α=2×10⁴ cm⁻¹ and α'=1×10⁴ cm⁻¹. In both curves, L and D are taken as 0.005 cm and 0.04 cm²/s, respectively. Note that the scale changes for the late decay in the inset.

which is precisely the form we have used in our earlier[35] PPTR work for obtaining absolute values of α at the excitation wavelength for "semi-infinite" samples.

An excitation spectrum of a distant sample can be obtained with back-scattered PPTR by scanning the wavelength of the excitation laser and monitoring the corresponding peak magnitude of the back-scattered PPTR signal. An example of such an excitation spectrum is shown in Fig. 5. Such an excitation spectrum truly represents an absorption spectrum of the sample under certain conditions, for example, when sample thickness is large and $\alpha' >> \alpha$; in this case Eq. (3) indicates that the peak value of $S_B(t)$ occurring at $t=0$ is proportional to $\alpha(\lambda)$ at the excitation wavelength λ.
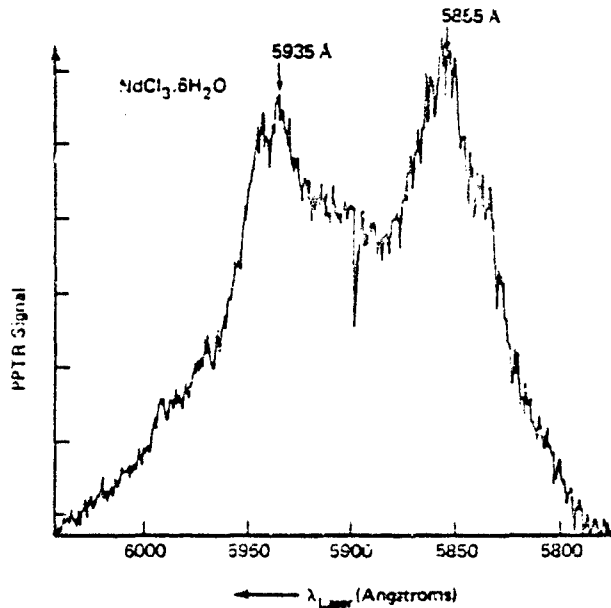

Fig. 5. PPTR excitation spectrum with back-scattering detection for $NdCl_3 6H_2O$. This is obtained by scanning the excitation pulsed dye laser over the Rhodamine 6G tuning range (5770Å to 6030Å). The numbers marked are in Angstrom units.

CONCLUSIONS

Techniques of PTR rely on the detection of modulated infrared thermal radiation from a sample that is excited by a modulated beam of energy. The recent developed technique of pulsed PTR in back-scattering detection is suitable for remote sensing. Its theoretical basis is discussed, and its new applications are indicated, which include absolute absorption spectroscopy, excitation spectroscopy, thickness or thermal diffusivity measurements, monitoring of layered structure, and sensing of degree of contact between materials. These applications should be valuable in science, technology, defense, and medical diagnostics (e.g., fast noncontact monitoring of skin in-vivo).

ACKNOWLEDGMENTS

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